

Organization **TC1700** Bldg. **REMSEN**

U. S. DEPARTMENT OF COMMERCE
COMMISSIONER FOR PATENTS

P.O. BOX 1450

ALEXANDRIA, VA 22313-1450

IF UNDELIVERABLE RETURN IN TEN DAYS

OFFICIAL BUSINESS

AN EQUAL OPPORTUNITY EMPLOYER



UNITED STATES POSTAGE
U.S. OFFICIAL MAIL
PENALTY FOR
PRIVATE USE \$300
02 1A
0004205065 JUL 24 2006
MAILED FROM ZIP CODE 22314
\$01.590
PRINCE BOWES

☐ A ☐ INSUFFICIENT ADDRESS
☐ C ☐ ATTEMPTED NOT KNOWN
☒ S ☐ NO SUCH NUMBER STREET
☐ NOT DELIVERABLE AS ADDRESSED
☐ UNABLE TO FORWARD
☐ OTHER

RTS
RETURN TO SENDER

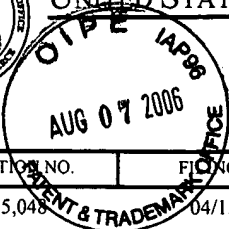
RECEIVED
AUG 07 2006
USPTO MAIL CENTER



UNITED STATES PATENT AND TRADEMARK OFFICE

LTZ

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
Address: COMMISSIONER FOR PATENTS
P.O. Box 1450
Alexandria, Virginia 22313-1450
www.uspto.gov



APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
10/825,048	04/15/2004	Areal Guerra Rogelio	G80-019 DIV	7402

7590

07/24/2006

NOTARO & MICHALOS P.C.
Empire State Building
Suite 6902
350 Fifth Avenue
New York, NY 10118-0110

EXAMINER

CORDRAY, DENNIS R

ART UNIT PAPER NUMBER

1731

DATE MAILED: 07/24/2006

Please find below and/or attached an Office communication concerning this application or proceeding.

Office Action Summary

Application No.

10/825,048

Applicant(s)

ROGELIO, AREAL GUERRA

Examiner

Dennis Cordray

Art Unit

1731

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address --

Period for Reply

A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE 3 MONTH(S) OR THIRTY (30) DAYS, WHICHEVER IS LONGER, FROM THE MAILING DATE OF THIS COMMUNICATION.

- Extensions of time may be available under the provisions of 37 CFR 1.136(a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.
- If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.
- Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133). Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).

Status

- 1) ☐ Responsive to communication(s) filed on ____.
- 2a) ☐ This action is **FINAL**. 2b) ☒ This action is non-final.
- 3) ☐ Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under *Ex parte Quayle*, 1935 C.D. 11, 453 O.G. 213.

Disposition of Claims

- 4) ☒ Claim(s) 1-6 is/are pending in the application.
- 4a) Of the above claim(s) ____ is/are withdrawn from consideration.
- 5) ☐ Claim(s) ____ is/are allowed.
- 6) ☒ Claim(s) 1-6 is/are rejected.
- 7) ☐ Claim(s) ____ is/are objected to.
- 8) ☐ Claim(s) ____ are subject to restriction and/or election requirement.

Application Papers

- 9) ☐ The specification is objected to by the Examiner.
- 10) ☐ The drawing(s) filed on ____ is/are: a) ☐ accepted or b) ☐ objected to by the Examiner.
Applicant may not request that any objection to the drawing(s) be held in abeyance. See 37 CFR 1.85(a).
Replacement drawing sheet(s) including the correction is required if the drawing(s) is objected to. See 37 CFR 1.121(d).
- 11) ☐ The oath or declaration is objected to by the Examiner. Note the attached Office Action or form PTO-152.

Priority under 35 U.S.C. § 119

- 12) ☒ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).
- a) ☒ All b) ☐ Some * c) ☐ None of:
1. ☐ Certified copies of the priority documents have been received.
 2. ☒ Certified copies of the priority documents have been received in Application No. 09744655.
 3. ☐ Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).

* See the attached detailed Office action for a list of the certified copies not received.

Attachment(s)

- 1) ☒ Notice of References Cited (PTO-892)
- 2) ☐ Notice of Draftsperson's Patent Drawing Review (PTO-948)
- 3) ☐ Information Disclosure Statement(s) (PTO-1449 or PTO/SB/08)
Paper No(s)/Mail Date ____.
- 4) ☐ Interview Summary (PTO-413)
Paper No(s)/Mail Date. ____.
- 5) ☐ Notice of Informal Patent Application (PTO-152)
- 6) ☐ Other: ____.

Art Unit: 1731

DETAILED ACTION

Specification

Applicant's claim for the benefit of a prior-filed application under 35 U.S.C. 119(e) or under 35 U.S.C. 120, 121, or 365(c) is acknowledged. Applicant has not complied with one or more conditions for receiving the benefit of an earlier filing date under 35 U.S.C. 120 as follows:

Applicant states that this application is a continuation or divisional application of the prior-filed application. A continuation or divisional application cannot include new matter. Applicant is required to change the relationship (continuation or divisional application) to continuation-in-part because this application contains the following matter not disclosed in the prior-filed application:

The prior-filed application, 09/744655, now U.S. Patent No.6743336, discloses preparation of a suspension of magnesium di-n-propylate from magnesium having a granulometric distribution lying between 50 and 150 m (col 5, lines 1-8 and 17-18; col 9, lines 13-15) whereas the instant Specification recites a corresponding granulometric distribution lying between 50 and 150 μ m (p 9, lines 19-26; p 10, lines 1-2; p 17, lines 33-35).

The disclosure is objected to because of the following informalities:

The word "CELLUSO" in the title of the invention should be changed to "CELLULOSIC" or "CELLULOSE."

On p 1, line 6, the missing U.S. Patent number 6,676,856 B1 should be filled in.

Appropriate correction is required.

Claim Rejections - 35 USC § 112

The following is a quotation of the first paragraph of 35 U.S.C. 112:

The specification shall contain a written description of the invention, and of the manner and process of making and using it, in such full, clear, concise, and exact terms as to enable any person skilled in the art to which it pertains, or with which it is most nearly connected, to make and use the same and shall set forth the best mode contemplated by the inventor of carrying out his invention.

Claim 5 is rejected under 35 U.S.C. 112, first paragraph, as failing to comply with the written description requirement. The claim(s) contains subject matter which was not described in the specification in such a way as to reasonably convey to one skilled in the relevant art that the inventor(s), at the time the application was filed, had possession of the claimed invention. Claim 5 recites magnesium powder having a granulometric distribution lying between 50 and 150 m. It is doubtful if magnesium particles of the claimed size exist or can even be manufactured.

The following is a quotation of the second paragraph of 35 U.S.C. 112:

The specification shall conclude with one or more claims particularly pointing out and distinctly claiming the subject matter which the applicant regards as his invention.

Claims 3-5 are rejected under 35 U.S.C. 112, second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which applicant regards as the invention.

Claim 3 recites reacting magnesium metal with n-propanol at "a boiling point temperature." Claim 5 recites cooling the reaction mixture to "a boiling point temperature." It is not clear what is meant by "a boiling point temperature" since several reactants, solvents and mixtures are potentially involved, each of which has a boiling point temperature different from any of the others. In addition, as the composition of the

Art Unit: 1731

reacting mixture changes, the boiling point temperature of the mixture will also change.

In the broadest sense of the term, almost any temperature of choice is a boiling point temperature for some substance or mixture of substances.

Claim 4 recites reacting magnesium metal with n-propanol at "a reflux temperature," with no indication of what reflux temperature is contemplated.

Claim 4 is also indefinite as to when the toluene is added to the mixture. Is it added prior to the reaction; to the reacting mixture, which will significantly change the "reflux temperature;" or after the reaction is essentially complete?

Claim 5 recites the step of "cooling said reaction mixture to a boiling point temperature when hydrogen is released." It is not clear when the reaction mixture is cooled. Is it cooled at the first indication of hydrogen release, after a pre-determined amount of hydrogen is released, or when the evolution of hydrogen ceases upon complete reaction?

Claim Rejections - 35 USC § 103

The following is a quotation of 35 U.S.C. 103(a) which forms the basis for all obviousness rejections set forth in this Office action:

(a) A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains. Patentability shall not be negated by the manner in which the invention was made.

Claims 1, 2 and 6 are rejected under 35 U.S.C. 103(a) as being unpatentable over Smith (6676856) or Worsfold (CA 2142195) in view of Smith (4860685).

Art Unit: 1731

Smith ('856) discloses method for deacidification of cellulosic materials by spraying with a composition comprising a carbonated magnesium alkoxide in a lower alcohol solvent, which has from 1 to 4 carbon atoms and a moisture content of less than 100 ppm. The method of making the carbonated magnesium alkoxide can comprise reacting magnesium metal in the form of metal chips (size unspecified) with a lower alcohol and carbon dioxide. The products have concentrations of 25 to 75%. (Abstract, col 1, lines 12-17; col 3, line 51 to col 4, line 24). Excess solvent is filtered out, leaving a deacidification concentrate, which is then diluted with an ultra dry solvent to make the deacidification composition (col 5, lines 13-21). The preferred diluent is HFC-134a (1,1,1,2-tetrafluoroethane) because the diluent has almost no detrimental affect on printing inks and forms stable compositions with the carbonated magnesium alkoxide (col 5, lines 50-54; col 6, lines 26-30). Smith ('856) teaches that it is known in prior art that moisture in anhydrous raw materials presents significant problems with known cellulose treatment methods (col 1, lines 38-53). Smith ('856) thus treats all solvents to reduce their moisture content prior to use (col 2, lines 51-64).

Worsfold discloses a composition for deacidification of cellulosic material comprising a carbonated magnesium alkoxide, an alcohol solvent, and hydrofluorocarbon diluent. The composition is applied by spraying the cellulosic material with the composition (Abstract). The method for production of methoxymagnesium methyl carbonate and ethoxymagnesium ethyl carbonate comprises reacting magnesium metal with methanol or ethanol to produce magnesium methoxide or magnesium ethoxide respectively and carbonation is performed by

Art Unit: 1731

treating a mixture of the methoxide in methanol or ethoxide in ethanol with carbon dioxide (p 5, line 34 to p 6, line 8). Worsfold discloses that other carbonated magnesium alkoxides can be prepared using other appropriate solvents to produce the corresponding homologs (p 6, lines 12-16). Suitable diluents include fluorinated ethanes and propanes (p 6, lines 23-27; p 7, lines 13-14).

Smith ('856) and Worsfold do not specifically disclose propoxymagnesium propyl carbonate (carbonated magnesium di-n-propylate) as a deacidification agent, although Worsfold does disclose that other carbonated magnesium alkoxides can be used.

Smith ('685) discloses a method for deacidification of cellulosic materials by spraying with a composition comprising a carbonated magnesium alkoxide (methoxymagnesium methyl carbonate, ethoxymagnesium ethyl carbonate and propoxymagnesium propyl carbonate are given as suitable examples), a lower alcohol solvent having from 1 to 3 carbon atoms and a chlorofluorocarbon diluent (Abstract, col 5, lines 13-35; col 6, lines 42-49).

The art of Smith ('856), Worsfold, Smith ('685) and the instant invention are analogous as pertaining to making and using deacidification compositions for cellulosic materials. It would have been obvious to one of ordinary skill in the art to use propoxymagnesium propyl carbonate as the deacidification agent in the composition of Smith ('856) or Worsfold in view of Smith ('685) as a functionally equivalent option. Using a dry carbon dioxide would be obvious to prevent unwanted side reactions per the teaching of Smith ('856). Note that Smith ('856) is usable as a reference under 35 U.S.C. 102(e) and thus is also usable under 35 U.S.C. 103(a).

Claims 2-5 are rejected under 35 U.S.C. 103(a) as being unpatentable over Smith (6676856) or Worsfold (CA 2142195) in view of Smith (4860685), as applied above to Claims 1 and 6, and further in view of Bell et al (3706665) and Sullivan (2265220).

Smith ('856), Worsfold, and Smith ('685) do not disclose reacting magnesium with propanol in the presence of iodine at a boiling point temperature. Smith ('856), Worsfold, and Smith ('685) also do not disclose using azeotropic distillation to remove excess propanol.

Bell et al disclose a process for preparing magnesium alkoxides, including magnesium n-propoxide that comprises reacting clean magnesium metal with excess alkanol (n-propanol for the propoxide) and using iodine as a catalyst (col 2, lines 24-32). Toluene can be added (col 2, lines 34-37). The reaction can be conducted at the refluxing temperature, which is a boiling temperature of the mixture, or can be cooled to moderate the reaction (col 2, lines 45-51). The reaction product of magnesium alkoxide can be carbonated by treating the solution with gaseous carbon dioxide to produce a product at least 50%, and preferably at least 60% carbonated (col 2, lines 52-71). Excess alcohol can be removed by azeotropic distillation (col 3, lines 25-30). Although an azeotrope between the propanol and toluene is not specifically disclosed by Bell et al, the presence of toluene in the mixture would form the azeotrope during the distillation process. Azeotropic separation is well known, as taught by Sullivan (p 1,

Art Unit: 1731

right col, lines 20-35). Sullivan also teaches that toluene and propanol form a low boiling azeotrope.

The art of Smith ('856), Worsfold, Smith ('685), Bell et al, Sullivan and the instant invention are analogous as pertaining to preparing magnesium alkoxides. It would have been obvious to one of ordinary skill in the art to use iodine as a catalyst in preparing the magnesium alkoxides in the process of Smith ('856) or Worsfold in view of Smith ('685) and further in view of Bell et al and Sullivan as a method known in the art of initiating the reaction. It would also have been obvious to employ azeotropic separation using toluene to remove the excess alcohol as a functionally equivalent option and well known technique in the art. Refluxing the reacting mixture would have been obvious to avoid loss of solvent during the reaction.

Conclusion


The prior art made of record and not relied upon is considered pertinent to applicant's disclosure [Kameinski et al (5208072) and Kameinski (4634786)]. They pertain to carbonated magnesium alkoxyalkyl carbonates used for deacidification of paper.

Any inquiry concerning this communication or earlier communications from the examiner should be directed to Dennis Cordray whose telephone number is 571-272-8244. The examiner can normally be reached on M - F, 7:30 -4:00 PM.

Art Unit: 1731

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Steven Griffin can be reached on 571-272-1189. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see <http://pair-direct.uspto.gov>. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free). If you would like assistance from a USPTO Customer Service Representative or access to the automated information system, call 800-786-9199 (IN USA OR CANADA) or 571-272-1000.


DRC
STEVEN P. GRIFFIN
SUPERVISORY PATENT EXAMINER
TECHNOLOGY CENTER 1700

Notice of References Cited	Application/Control No. 10/825,048		Applicant(s)/Patent Under Reexamination ROGELIO, AREAL GUERRA	
	Examiner Dennis Cordray		Art Unit 1731	Page 1 of 1

U.S. PATENT DOCUMENTS

*		Document Number Country Code-Number-Kind Code	Date MM-YYYY	Name	Classification
*	A	US-2,265,220 A	12-1941	SULLIVAN JR FREDERICK W	203/54
*	B	US-3,706,665 A	12-1972	Bell et al	508/358
*	C	US-4,634,786 A	01-1987	Kamienski, Conrad W.	556/187
*	D	US-4,860,685 A	08-1989	Smith, Richard D.	118/300
*	E	US-5,208,072 A	05-1993	Kamienski et al.	427/296
*	F	US-6,676,856 B1	01-2004	Smith, Richard Daniel	252/387
	G	US-			
	H	US-			
	I	US-			
	J	US-			
	K	US-			
	L	US-			
	M	US-			

FOREIGN PATENT DOCUMENTS

*		Document Number Country Code-Number-Kind Code	Date MM-YYYY	Country	Name	Classification
	N	CA 2142195 A1	08-1996	CANADA	WORSFOLD	
	O					
	P					
	Q					
	R					
	S					
	T					

NON-PATENT DOCUMENTS

*		Include as applicable: Author, Title Date, Publisher, Edition or Volume, Pertinent Pages)
	U	
	V	
	W	
	X	

*A copy of this reference is not being furnished with this Office action. (See MPEP § 707.05(a).)
Dates in MM-YYYY format are publication dates. Classifications may be US or foreign.



(21) (A1)

2,142,195

(22)

1995/02/08

(43)

1996/08/09

(51) Int.Cl. ⁶ D21H 25/18

(19) (CA) APPLICATION FOR CANADIAN PATENT (12)

(54) Deacidification of Cellulosic Material

(72) Worsfold, D. James - Canada ;

(71) Same as inventor

(57) 20 Claims

Notice: This application is as filed and may therefore contain an incomplete specification.



DEACIDIFICATION OF CELLULOSIC MATERIAL

ABSTRACT

A composition for deacidification of aging cellulosic materials, such as paper, books, documents, works of art, clothing and flags, from library, archive and museum collections is comprised of a carbonated magnesium alkoxide, such as methoxymagnesium methylcarbonate or ethoxymagnesium ethylcarbonate, a solvent, such as methanol or ethanol, and a hydrochlorofluorocarbon and/or a hydrofluorocarbon diluent. The deacidification composition of the present invention may be applied by spraying through a nozzle, by brushing or by immersing the cellulosic material in the deacidification composition.

DEACIDIFICATION OF CELLULOSIC MATERIAL

FIELD OF THE INVENTION

The present invention relates to the field of
5 preservation of cellulosic materials, such as books,
documents, works of art, clothing and flags, and in
particular, to a process for deacidification of cellulosic
materials.

10 BACKGROUND OF THE INVENTION

There is much concern about the deterioration of aging
cellulosic materials, particularly those of library, museum
and archive collections. The most significant cause of
deterioration of cellulosic materials is acid-catalyzed
15 hydrolysis of cellulose which results in shortened
cellulose molecules, and weak and brittle fibres. There
are a number of factors which cause an acidic condition in
cellulosic materials including air pollutants, the use of
alum in the paper-making process and the degradation
20 products of cellulose and ink.

In an effort to reduce the deterioration of cellulosic
materials by acid-catalyzed hydrolysis, researchers have
turned their attention to processes for deacidification of
cellulosic materials. Attention has been specifically
25 directed to non-aqueous deacidification compositions to
neutralize the acidity of the cellulosic materials and to
introduce an alkaline reserve to inhibit re-acidification.

United States Patent Number 3,676,182 (R.D. Smith,
July 11, 1972) relates to a process for non-aqueous
30 deacidification of paper using magnesium methoxide as a
deacidification agent. The deacidification agent is
dissolved in methanol to a concentration of about 5 to 11%.
The resultant solution may be diluted with a
chlorofluorocarbon, such as trichlorofluoromethane or
35 dichlorodifluoromethane, to produce a solution of about 1
to 2% magnesium methoxide. The chlorofluorocarbon
contributes to rapid evaporation of the solution and

imparts hydrophobic properties to deacidification solutions containing methanol, thereby minimizing attack on certain inks by methanol. Books and papers may be dipped in the solution or the solution may be applied by brushing or spraying.

However, magnesium methoxide is extremely sensitive to water such that even traces of moisture cause immediate hydrolysis and a gelatinous precipitate of magnesium hydroxide which is insoluble in water and inorganic solvents. Unless the paper is sufficiently dried prior to treatment, premature hydrolysis of the magnesium methoxide may occur, causing an undesirable glaze of magnesium hydroxide to be formed on the surface of the paper. Furthermore, the nozzles of spray guns are subject to frequent plugging. The bristles of brushes also become clogged and must be cleaned and dried prior to reuse.

United States Patent Number 3,939,091 (Kelly, G.B., February 17, 1976) discloses a composition for use in the deacidification of paper which overcomes the problem of the prior art deacidification solutions comprised of magnesium methoxide dissolved in a chlorofluorocarbon. Magnesium methoxide is dissolved in methanol or in a mixture of methanol and trichlorotrifluoroethane (FREONTM TF). Carbon dioxide is then introduced into the solution to produce a colourless 20% methoxymagnesium methylcarbonate solution. The resultant solution is very tolerant of water compared to solutions of magnesium methoxide and does not cause plugging of spray nozzles or gumming of brushes.

Canadian Patent Number 1,147,510 (Smith, R.D., June 7, 1983) describes another method for production of methoxymagnesium methylcarbonate. Magnesium metal is reacted with absolute methanol to produce dried magnesium methoxide. The magnesium methoxide is then re-dissolved in methanol containing carbon dioxide to produce a solution of methoxymagnesium methylcarbonate. The solution is diluted with trichlorotrifluoroethane or dichlorodifluoromethane. The diluted solution is sprayed or brushed onto papers to

deacidify the paper. Alternatively, the paper can be dipped into the solution. This method for preparation of methoxymagnesium methylcarbonate allows the dry material, namely magnesium methoxide, to be prepared prior to
5 delivery to a work site and then re-dissolved in methanol and reacted with carbon dioxide on site.

United States Patent Number 4,860,685 (Smith, R.D., August 29, 1989) and corresponding Canadian Patent Number 1,272,018 (July 31, 1990) describe a soft spray system for
10 deacidification of cellulosic materials. A deacidification agent, preferably a carbonated magnesium alkoxide, is combined with a chlorofluorocarbon diluent, preferably trichlorotrifluoroethane, and a chlorofluorocarbon gas propellant, preferably dichlorodifluoromethane. Additional
15 pressurizing and propelling may be provided by an inert gas such as nitrogen. The carbonated magnesium alkoxide, such as methoxymagnesium methylcarbonate or ethoxymagnesium ethylcarbonate, is produced by dissolving the corresponding magnesium alkoxide in a lower alcohol in the presence of
20 carbon dioxide.

The prior art processes described herein use chlorofluorocarbons since they are substantially unreactive and, as such, pose no direct toxic threat to living organisms. However, these very same characteristics that
25 render chlorofluorocarbons inert pose significant problems in the stratosphere. Chlorofluorocarbons have been recognized as being a major factor responsible for stratospheric ozone depletion and for contributing to the greenhouse effect. Of particular concern is the chlorine
30 which is liberated when the chlorofluorocarbons are exposed to strong UV radiation in the stratosphere. Chlorine depletes ozone by catalyzing its conversion to molecular oxygen. Production and consumption of chlorofluorocarbons has been substantially reduced and must be eliminated by
35 January 1, 1996 (Copenhagen Agreement to amend Montreal Protocol, November 23-25, 1992). Accordingly, a substitute must be found for the chlorofluorocarbon diluent of the

prior art compositions.

It is an object of the present invention to provide a composition for deacidification of aging cellulosic material comprising a diluent to replace the
5 chlorofluorocarbon diluents of the prior art.

SUMMARY OF THE INVENTION

According to one aspect of the present invention, there is provided a composition for deacidification of a
10 cellulosic material, comprising a carbonated magnesium alkoxide, a solvent, and a hydrochlorofluorocarbon and/or a hydrofluorocarbon diluent.

According to another aspect of the present invention, there is provided a method for preparing a composition for
15 deacidification of a cellulosic material, comprising the steps of refluxing magnesium metal in methanol to produce a suspension of magnesium methoxide in methanol; cooling the suspension of magnesium methoxide in methanol; saturating the cooled suspension of magnesium methoxide in
20 methanol with carbon dioxide to produce a solution of methoxymagnesium methylcarbonate in methanol; and diluting the solution of methoxymagnesium methylcarbonate with a hydrochlorofluorocarbon or hydrofluorocarbon diluent.

25 DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

In accordance with the present invention, the chlorofluorocarbons of the prior art deacidification compositions are replaced with a hydrochlorofluorocarbon and/or a hydrofluorocarbon.

30 The present inventor has discovered that carbonated magnesium alkoxide deacidification agents are soluble in hydrochlorofluorocarbons and/or hydrofluorocarbons and that the resultant compositions are effective in deacidification of cellulosic materials. Moreover,
35 hydrochlorofluorocarbons and hydrofluorocarbons are substantially inert and are not toxic.

The use of hydrochlorofluorocarbons and

hydrofluorocarbons to replace the chlorofluorocarbons of the prior art compositions substantially reduces the available number of chlorine ions which may be liberated upon exposure to strong UV radiation in the stratosphere, as compared with the prior art chlorofluorocarbons. Furthermore, the presence of hydrogen renders the compound more reactive so that it is more likely to be decomposed before it reaches the stratosphere. Moreover, the rate of decomposition is higher than that of chlorofluorocarbons which have a lifetime of about 60 to 100 years. While regulations will eventually prohibit the use of hydrochlorofluorocarbons, the phase-out schedule for consumption only begins on January 1, 1996 with elimination scheduled for 2030 (Copenhagen Agreement to amend Montreal Protocol, November 23-25, 1992). There are no such regulations in place with respect to hydrofluorocarbons.

The hydrochlorofluorocarbon and/or hydrofluorocarbon act as a diluent for delivery of a deacidification agent and a solvent to aging cellulosic materials. Hereinafter, the term "cellulosic material" will be understood to include paper, books, documents, archival records, maps, works of art and articles made from cotton and/or linen including clothing and flags.

A suitable deacidification agent is a carbonated magnesium alkoxide, such as methoxymagnesium methylcarbonate or ethoxymagnesium ethylcarbonate.

One method for production of methoxymagnesium methylcarbonate is described in United States Patent Number 3,939,091. Magnesium metal is refluxed in methanol to give a suspension of magnesium methoxide in methanol. After cooling, the suspension of magnesium methoxide in methanol is saturated with carbon dioxide to form a solution of methoxymagnesium methylcarbonate in methanol.

Methoxymagnesium methylcarbonate and ethoxymagnesium ethylcarbonate may also be produced as described in Canadian Patent Number 1,147,510 wherein magnesium metal is reacted with methanol or ethanol to produce magnesium

methoxide or magnesium ethoxide, respectively. The resultant solution is dried, by a method known to those skilled in the art, to form a dried powder of magnesium alkoxide. The dried magnesium methoxide or magnesium ethoxide is subsequently added to methanol or ethanol in the presence of carbon dioxide to form methoxymagnesium methylcarbonate or ethoxymagnesium ethylcarbonate, respectively.

Preferably, the carbonated magnesium alkoxide is methoxymagnesium methylcarbonate or ethoxymagnesium ethylcarbonate, with methoxymagnesium methylcarbonate being the most preferable. However, it will be appreciated by those skilled in the art that other suitable carbonated magnesium alkoxides can be prepared by either of the above-mentioned methods using other solvents to produce the corresponding homologs of methoxymagnesium methylcarbonate. The solvent must however be evaluated for the potential for damage to the bindings, inks, etc. of the material being treated.

The concentration of carbonated magnesium alkoxide in the solvent is suitably in the range of from about 5 to 20% (w/v). Preferably, the concentration is about 20% (w/v).

The resultant solution of carbonated magnesium alkoxide in solvent is diluted with a hydrochlorofluorocarbon and/or a hydrofluorocarbon to yield a concentration of carbonated magnesium alkoxide in the range of from about 0.1 to 2% (w/v). Preferably, the concentration of carbonated magnesium alkoxide in the diluted solution is in the range of from about 0.5 to 1.5% (w/v). The hydrochlorofluorocarbon and/or hydrofluorocarbon diluent contributes to rapid evaporation of the solution and minimizes attack on certain inks by the solvent. It is desirable that the concentration of solvent in the deacidification composition of the present invention is as low as possible. Preferably, the concentration of solvent in the diluted solution is less than about 5% (v/v).

Suitable hydrochlorofluorocarbon diluents are difluorochloroethane, trifluorochloroethane, tetrafluorochloroethane, hydrochlorofluoropropanes and hydrochlorofluorobutanes. A particularly suitable

5 hydrochlorofluorocarbon diluent is 1,1-dichloro-1-fluoroethane. Chlorodifluoromethane may be added as a co-diluent to increase the vapour pressure of the mixture, thereby increasing evaporation of the solution. Preferably, the concentration of chlorodifluoromethane
10 added as a co-diluent is not greater than about 10% (v/v), in view of the high vapour pressure of chlorodifluoromethane.

Suitable hydrofluorocarbons are fluorinated methanes, ethanes, propanes and butanes.

15 The deacidification composition of the present invention may also include additives, such as a plasticizer, to reduce the brittleness of the cellulosic material.

The deacidification composition of the present
20 invention may be applied to cellulosic materials in a manner known to those skilled in the art, for example by spraying with a spray gun or an aerosol can, by brushing or by dipping the cellulosic materials into the deacidification composition.

25 Preferably, the method for deacidification of cellulosic materials is as follows.

The cellulosic materials are placed in a vessel capable of withstanding a vacuum. The vessel is sealed and evacuated with a vacuum pump. Preferably, the water
30 content of the cellulosic materials is reduced from a normal equilibrium moisture content of from about 5 to 6% to a residual moisture content of from about 1 to 2% under vacuum. The deacidification composition of the present invention is then introduced to the vessel and the
35 cellulosic materials are immersed in the composition for a time sufficient to allow penetration of the composition into the cellulosic materials.

If desired, a positive pressure, for example by introduction of carbon dioxide or another gas, may be applied to the vessel to increase the penetration of the composition.

5 The treatment may be conducted at room temperature or greater. It will be appreciated by those skilled in the art that the temperature must not be so great as to damage the cellulosic material being treated.

10 At the end of the treatment, the vessel is drained and a vacuum is applied to the vessel to recover any remaining solvent and diluent. The cellulosic material is then removed from the vessel and is allowed to air dry in a fumehood. This method is particularly advantageous in that it permits simultaneous treatment of a large number of
15 books, for example. Furthermore, the solution can be readily recovered from the vessel.

 Alternatively, the deacidification composition of the present invention may be applied by the soft spray system described in United States Patent Number 4,860,685,
20 discussed hereinabove. It will be appreciated by those skilled in the art that other methods of contacting the cellulosic materials with the deacidification composition of the present invention are also suitable. For example, it is not necessary that the cellulosic material be dried
25 under a vacuum prior to contacting with the deacidification composition of the present invention. The cellulosic material may also be contacted with the deacidification composition of the present invention by brushing or spraying.

30 The deacidification composition of the present invention may also be used in the method and apparatus described in Canadian Patent Application Number 2,009,621 (Eggersdorfer, R. et al, published August 11, 1990). Canadian Patent Application Number 2,009,621 relates to a
35 method and apparatus for deacidification of cellulosic materials by pre-drying the paper by high frequency radiation in a vacuum, neutralizing with a deacidification

solution and evaporating the solvent in a vacuum with high frequency radiation. The apparatus described is a single treatment chamber for the pre-drying, neutralizing and evaporating steps in an enclosure for total solvent recovery.

5

CLAIMS:

1. A composition for deacidification of a cellulosic material, comprising a carbonated magnesium alkoxide, a solvent, and a hydrochlorofluorocarbon and/or a hydrofluorocarbon diluent.
2. A composition according to claim 1, wherein the hydrochlorofluorocarbon diluent is selected from the group consisting of difluorochloroethane, trifluorochloroethane, tetrafluorochloroethane, hydrochlorofluoropropanes and hydrochlorofluorobutanes.
3. A composition according to claim 2, further comprising chlorodifluoromethane as a co-diluent.
4. A composition according to claim 3, wherein the concentration of chlorodifluoromethane is not greater than about 10% (v/v).
5. A composition according to claim 1, wherein the hydrofluorocarbon diluent is selected from the group consisting of fluorinated methanes, ethanes, propanes and butanes.
6. A composition according to claim 1, wherein the carbonated magnesium alkoxide is methoxymagnesium methylcarbonate.
7. A composition according to claim 1, wherein the carbonated magnesium alkoxide is ethoxymagnesium ethylcarbonate.
8. A composition according to claim 1, wherein the concentration of the carbonated magnesium alkoxide is in the range of from about 0.1 to 2% (w/v).
9. A composition according to claim 1, wherein the concentration of the carbonated magnesium alkoxide is in

the range of from about 0.5 to 1.5% (w/v).

10. A composition according to claim 1, wherein the solvent is methanol.

11. A composition according to claim 1, wherein the solvent is ethanol.

12. A composition according to claim 1, wherein the concentration of solvent is less than about 5% (v/v).

13. A method for preparing a composition for deacidification of a cellulosic material, comprising the steps of refluxing magnesium metal in methanol to produce a suspension of magnesium methoxide in methanol; cooling the suspension of magnesium methoxide in methanol; saturating the cooled suspension of magnesium methoxide in methanol with carbon dioxide to produce a solution of methoxymagnesium methylcarbonate in methanol; and diluting the solution of methoxymagnesium methylcarbonate with a hydrochlorofluorocarbon or hydrofluorocarbon diluent.

14. A method according to claim 13, wherein the hydrochlorofluorocarbon diluent is selected from the group consisting of difluorochloroethane, trifluorochloroethane, tetrafluorochloroethane, hydrochlorofluoropropanes and hydrochlorofluorobutanes.

15. A method according to claim 14, wherein chlorodifluoromethane is used as a co-diluent.

16. A method according to claim 15, wherein the concentration of chlorodifluoromethane is not greater than about 10% (v/v).

17. A method according to claim 13, wherein the hydrofluorocarbon diluent is selected from the group consisting of fluorinated methanes, ethanes, propanes and butanes.

18. A method according to claim 13, wherein the concentration of methoxymagnesium methylcarbonate in the diluted composition is in the range of from about 0.1 to 2% (w/v).

19. A method according to claim 13, wherein the concentration of methoxymagnesium methylcarbonate in the diluted composition is in the range of from about 0.5 to 1.5% (w/v).

20. A method according to claim 13, wherein the concentration of methanol in the diluted composition is less than about 5% (v/v).

**This Page is Inserted by IFW Indexing and Scanning
Operations and is not part of the Official Record**

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- ☐ BLACK BORDERS
- ☒ IMAGE CUT OFF AT TOP, BOTTOM OR SIDES
- ☒ FADED TEXT OR DRAWING
- ☐ BLURRED OR ILLEGIBLE TEXT OR DRAWING
- ☐ SKEWED/SLANTED IMAGES
- ☒ COLOR OR BLACK AND WHITE PHOTOGRAPHS
- ☐ GRAY SCALE DOCUMENTS
- ☒ LINES OR MARKS ON ORIGINAL DOCUMENT
- ☐ REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY
- ☐ OTHER: _____

IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.